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In situ laser irradiation setup for a Bruker three-circle goniometer

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A new design of a setup for *in situ* laser irradiation of single crystals during an X-ray diffraction experiment is presented. The system is designed for use with a Bruker three-circle goniometer in combination with a Helix ultra-low-temperature cryostat and consists of a laser mount and a set of three adjustable mirrors. The main advantages of the presented system include a stationary laser mount, the ability to irradiate a sample inside the Be nozzle and no impediments to the goniometer movements.

1. Introduction

Photocrystallographic experiments have remained a focus of research groups for many years. Such studies provide ample information about photochemical processes in the solid state, be it about light-induced chemical transformations (Chaudhary *et al.*, 2017), structures of exited states (Raithby, 2015; Hatcher & Raithby, 2013) or spin-crossover phase transitions (Létard *et al.*, 2012). To the best of our knowledge in the vast majority of such studies, especially those related to time-resolved crystallography, synchrotron sources were used (Fullagar *et al.*, 2000). The mutual orientation of low-temperature (LT) devices, the primary beam and goniometers in a typical synchrotron setup is quite different from that of an ordinary X-ray installation, and therefore the engineering solutions applied there are rarely transferable to the laboratory. When irradiation took place in a laboratory environment, it was usually performed in a stationary position and not simultaneously with data collection (see *e.g.* Casaretto *et al.*, 2015).

The continued interest in these sorts of studies has resulted in a demand for laboratory-based instruments able to irradiate a crystal during data collection and operating at very low temperatures, meaning the presence of an He open-flow cryostat. One of the first such devices was designed more than a decade ago at Durham University (Thompson *et al.*, 2004) and was successfully used for a number of years [some results are reported by Létard *et al.* (2012)]. The setup consisted of a laser and a set of mirrors mounted on the 2θ arm of a Bruker three-circle diffractometer equipped with a Helix (Oxford Cryosystems) cryostat. The advantages of the setup included the possibility of the fine alignment of the laser beam and irradiation of the crystal from beneath, thus allowing irradiation of a sample hidden inside of the nozzle of the LT device. However, the apparatus had its shortcomings; in particular, moving the laser together with the detector arm required very careful positioning of the laser's power supply unit (and connected wires) in order to avoid obstruction of the goniometer movements due to entanglement of the wires. Additional weight of the laser and mirrors could also result in excessive wear of the 2θ gear in the long term. As mentioned, the system was successfully used for a number of years and only the replacement of an old diffractometer by a new one forced the author to re-design the irradiation system. The main reason for re-design was the introduction by Bruker of a motorized detector track that made the mounting of a laser support unit on the detector arm quite awkward.

It should be noted that recently another quite ingenious device, serving the very same purpose, has been reported (Kamiński *et al.*, 2016). The authors used a fibre-optic cable to transfer the laser light from a standalone light source to the focusing unit, mounted next to the X-ray source on a specially designed support block. This system provided a number of advantages (these include a perfect beam stability and high flexibility in the choice of light sources). However, one of the features of that apparatus probably makes it impossible to use in combination with the Helix LT device: in the configuration described in this paper, the horizontal beam would be blocked by the Be nozzle of the Helix device. At the same time the beam would be obstructed by the ω block if it were moved to a lower position where the light beam would pass below the edge of the nozzle.

This made that device unsuitable for Helix-based low-temperature studies and a new system had to be developed. The new design was supposed to keep all the advantages of the old model but add some new features, such as a laser mount independent of the goniometer. This should provide improved stability for the laser beam, more choice in positioning of the laser's power supply unit and the absence of any interference with the goniometer movements. In this short paper this new design is described.

2. Design

The new *in situ* irradiation device was intended to be used on a Bruker D8 Venture dual IμS source diffractometer with a motorized Photon-100 CMOS detector and equipped with long-nose Helix and Cryostream-700 (both Oxford Cryosystems) cryostats. In this instrument configuration the Cryostream support block is fixed to the ceiling of the goniometer safety enclosure, but the massive stand for the Helix is placed around three sides of the goniometer. All these units together with associated pipes and cables create a rather overcrowded crystal (and goniometer) environment.

The design of the new device is shown in Fig. 1.

Several considerations have been taken into account during the design of the new system. The most important requirement was the direction of the light beam: in order to illuminate the sample inside the nozzle the beam should come from beneath. All the designs involving the mounting of any fibre-optic cables underneath the optical centre of the goniometer were deemed to be unfeasible owing to the confined space and the presence of moving parts. So, apparently the most sensible solution was to place a mirror (M3) on the flat part of the ω circle beneath the crystal and choose the right position of the light source in order to reflect the beam onto the crystal. The test runs showed that during the rotation of the ω circle the position of M3 was unchanged, no vibration was noticed and thus the position of the beam during goniometer movement was stable. The introduction by Bruker of the shutterless data collection mode, where the ω circle moves at constant speed during the data collection, enhanced the stability of the reflected laser beam even further. M3 is kept in place by three small magnets and is easily removable during routine day-to-day operations.

After careful examination of possible positions of the incoming beam, the geometry where the beam is passing through a gap between the Helix unit and the detector was chosen. The corresponding adjustable mirror (M2) was mounted at the chosen spot on the ceiling of the safety enclosure. The mount is attached to the enclosure by several small magnets, and that provides a solid contact but at the same time makes the position of mirror M2 easily changeable. In order to keep the beam coming from mirror M2 on the crystal, the reflection point on mirror M3 should be located slightly out of the centre of the ω circle. This means that during data collection the reflection point traces a short curve on the surface of the ω circle. It turned out that a flat mirror of 80 mm diameter is more than sufficient to cover the length of the curve and therefore such a mirror was used as M3.

The following considerations were kept in mind when choosing the position of the light source: the laser mount

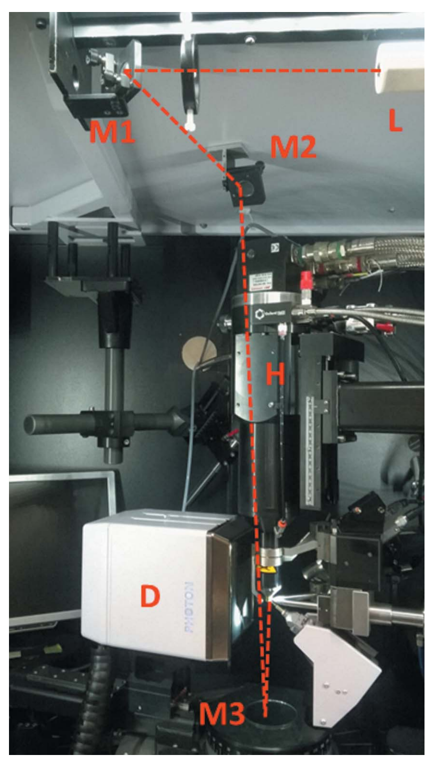

Figure 1
A view of the device on the Bruker D8 Venture diffractometer. The beam path is shown as a red dashed line. Abbreviations: L – a laser; M1, M2, M3 – mirrors; H – the Helix LT device; D – the Photon-100 detector.

should not be connected to the goniometer (for the sake of mechanical stability), it should be easily accessible, and it should not cause any obstructions for everyday standard data collection procedures and for the maintenance of the instrument.

These somewhat contradictory requirements were accommodated by mounting the laser on the ceiling of the safety enclosure just behind the front panel. The previously used (Thompson *et al.*, 2004) guide rings with attached adjustable mirror (M1) were scavenged to mount the light source. This position of the light source enables easy access for focusing of the beam and maintenance and also leaves plenty of options for positioning of the power supply units (providing the cables are sufficiently long) inside the safety enclosure. At the same time the ceiling mounting does not obstruct access to the goniometer and does not interfere with the goniometer movement. The positioning of the laser in place of M2 would make the beam alignment procedure more awkward because it is much easier to adjust the position of a small mirror rather than a whole laser mount. The pointing of the laser directly onto M2 would also require placing the laser almost perpendicular to the front panel of the safety enclosure. Such an orientation would reduce the space available for the laser mount and consequently limit the maximum size of the light source.

The focusing of the laser beam on the crystal is achieved by the fine combined adjustments of the positions of mirrors M1 and M2. The test data collections confirmed the high stability of the beam on the crystal and the ease of the beam-focusing procedure.

3. Conclusions

A new device for *in situ* irradiation of a single crystal on a Bruker three-circle goniometer has been designed. The device comprises a light-source mount and three mirrors, one of which is located on the surface of the ω circle underneath the crystal. The other two mirrors and the light source are attached to the ceiling of the safety enclosure, thus providing unobstructed access to the goniometer. The use of magnetic mounts and finely adjustable mirrors makes the system flexible/removable and the focusing procedure straightforward. The device provides constant irradiation of the crystal during the data collection and may be used with a Helix cryostat, when the crystal is blocked from the side view by the Be nozzle.

References

Casaretto, N., Pillet, S., Bendeif, E. E., Schaniel, D., Gallien, A. K. E., Klüfers, P. & Woike, T. (2015). *IUCrJ*, **2**, 35–44.

Chaudhary, A., Mohammad, A. & Mobin, S. M. (2017). *Cryst. Growth Des.* **17**, 2893–2910.

Fullagar, W. K., Wu, G., Kim, C., Ribaud, L., Sagerman, G. & Coppens, P. (2000). *J. Synchrotron Rad.* **7**, 229–235.

Hatcher, L. E. & Raithby, P. R. (2013). *Acta Cryst.* C**69**, 1448–1456.

Kamiński, R., Jarzembska, K. N., Kutyła, S. E. & Kamiński, M. (2016). *J. Appl. Cryst.* **49**, 1383–1387.

Létard, J.-F., Asthana, S., Shepherd, H. J., Guionneau, P., Goeta, A. F., Suemura, N., Ishikawa, R. & Kaizaki, S. (2012). *Chem. Eur. J.* **18**, 5924–5934.

Raithby, P. R. (2015). *IUCrJ*, **2**, 5–6.

Thompson, A. L., Beeby, A. & Goeta, A. E. (2004). *J. Appl. Cryst.* **37**, 652–653.